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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
09/544,344	04/06/2000	Arthur W. Snow	0064612-0010	8024

7590 09/02/2003

Associate Counsel (Patents)  
Code 10082  
Naval Research Laboratory  
Washington, DC 20375-5000

EXAMINER

SODERQUIST, ARLEN

ART UNIT	PAPER NUMBER
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1743

DATE MAILED: 09/02/2003

17

Please find below and/or attached an Office communication concerning this application or proceeding.

# Office Action Summary

Application No.

09/544,344

Applicant(s)

SNOW ET AL.

Examiner

Arlen Soderquist

Art Unit

1743

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

## Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

## Status

- 1) ☒ Responsive to communication(s) filed on 04 June 2003.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

## Disposition of Claims

- 4) ☒ Claim(s) 1-9 and 21-36 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1-9 and 21-34 is/are rejected.
- 7) ☒ Claim(s) 35 and 36 is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

## Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
- Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
- 11) ☐ The proposed drawing correction filed on \_\_\_\_\_ is: a) ☐ approved b) ☐ disapproved by the Examiner.
- If approved, corrected drawings are required in reply to this Office action.
- 12) ☐ The oath or declaration is objected to by the Examiner.

## Priority under 35 U.S.C. §§ 119 and 120

- 13) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some \* c) ☐ None of:
- ☐ Certified copies of the priority documents have been received.
  - ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
  - ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).
- \* See the attached detailed Office action for a list of the certified copies not received.
- 14) ☒ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. § 119(e) (to a provisional application).
- a) ☐ The translation of the foreign language provisional application has been received.
- 15) ☒ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. §§ 120 and/or 121.

## Attachment(s)

- 1) ☒ Notice of References Cited (PTO-892)
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) ☐ Information Disclosure Statement(s) (PTO-1449) Paper No(s) \_\_\_\_\_.
- 4) ☐ Interview Summary (PTO-413) Paper No(s). \_\_\_\_\_.
- 5) ☐ Notice of Informal Patent Application (PTO-152)
- 6) ☐ Other: \_\_\_\_\_.

Art Unit: 1743

1. Applicant's arguments in the Appeal Brief raised questions that were not adequately responded to in the rejection and response of the last Office action, therefore, to properly address those questions and to include newly discovered problems with the application, the finality of that action is withdrawn.

2. The following is a quotation of the first paragraph of 35 U.S.C. 112:

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same and shall set forth the best mode contemplated by the inventor of carrying out his invention.

3. Claims 1-9 and 27-28 are rejected under 35 U.S.C. 112, first paragraph, because the specification, while being enabling for methods of fabricating an assembly for investigation of a target environment to determine the presence of a target species or an article of manufacture formed by the methods disclosed on page 17, line 8 to page 19, line 2 page 22 lines 9-18 and examples 26-30, does not reasonably provide enablement for the scope found in claims 1-9 and 27-28. The specification does not enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the invention commensurate in scope with these claims. First with respect to claims 1-9 and 27, page 17, lines 8-12 teach

"Formation of the multiplicity of particles as a thin film is a sensitive operation. In our experience, casting from solution with slow evaporation does not produce a thin film with reproducibility or acceptable uniformity. We have invented various methods that address this problem."

Thus all methods of forming the composition do not result in an acceptable film or multiplicity of particles and the specification fails to enable a claim of the scope found in claims 1-9 and 27. with respect to claim 28 the claim language does not find antecedent basis in the specification.

Page 22 lines 9-16 teach

"MIME sensor 64 comprises particles of gold core having a nominal radius of 1 nanometer combined with an alkanethiol ligand shell having 8 carbon atoms in the alkane chain. A film of this core-ligand material approximately 0.4 micrometers in thickness was sprayed onto a small interdigital electrode component consisting of 50 "finger pairs" of gold 15  $\mu\text{m}$  wide and spaced 15  $\mu\text{m}$  from the next element to form sensor 64."

This in combination with the process of making the particles as found on pages 13-14 of the instant specification regarding the need for a reducing agent and rapid mixing show that a

Art Unit: 1743

solution containing the core-ligand particles is first formed and then sprayed onto the electrode. Thus the process of claim 28 is not supported by the specification.

4. In the rejections that follow, examiner will be citing portions of the instant specification to show that various claimed properties are inherent in the teachings of the applied references. The format used will be bold italicized letters contained within parentheses.

5. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless –

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

6. Claims 1- 5, 7 - 9, 21- 27 and 33 - 34 are rejected under 35 U.S.C. 102(b) as being anticipated by Bethell (*J. Electroanal. Chem.*). In the paper Bethell describes simple methods for the production of Au nanoparticles with narrow size distributions by reduction of tetrachloroaurate solutions in the presence of thiol-containing organic compounds which self-assemble on the Au surface. Stable solutions of somewhat larger particles can be produced if the thiol is absent. The thiol-derivatized materials are stable in air over long periods and can be handled in much the same way as simple organic compounds. Page 138, column 1, last full paragraph teaches nonanethiol derivatized particles having a core diameter of 1.5-3 nm. Subheading 2.1 contains a reference to an article by M. Brust which is found cited in the instant specification as one of the methods for making the metal core-ligand particles of the instant invention(*see page 13, line 23 to page 14, line 8*). In figure 1(b) a spectrum of dodecanethiol-derivatized particles is presented (*see examples 10-13, page 33 line 9 to page 34, line 10 for a description of particles made with dodecanethiol*). Table 1 teaches a range of dithiol containing molecules. Each of these is clearly within the thiol derivatives taught in the instant specification (*see page 12 line 21 to page 13 line 11 and 26-30 page 39, line 12 to page 41, line 23*). Using dithiols as the derivatizing spacer units, methods were developed for the preparation of materials in 3-dimensional form and as thin films attached to a solid substrate (figure 3, and the only full paragraph on page 139). (*A comparison of this procedure with that found in examples 26-30 shows that there is significant similarity and that the Bethell process forms a self-assembled layered structure as in the examples.*) Such materials show conductivities that

Art Unit: 1743

mimic the behavior of semiconductors and that depend markedly on the structure of the dithiol used to link the Au particles together. Thus there is inherently a structure capable of being used to measure conductivity. The increase in conductivity with increasing temperature probably involves activated electron hopping from particle to particle. Surfaces treated with a coating of the materials show electroreflectance changes with applied potential that also differ according to the structure of the dithiol spacer. Unusual effects were observed on heterogeneous electron transfer from electrode surfaces treated with layers of the Au nanoparticles and dithiol spacers. Applications for these nanostructured materials can be envisaged, which range from submicroelectronic devices and circuitry to electrical modification of the reflectance of glass. Such applications will require a multidisciplinary approach with a substantial organic chemical research input. Section 4 and figure 5 discuss other methods of making the clusters which use multi-functional groups on the ligand layer surrounding the metal core. *(Additional sections of the instant specification that are relevant to the inherency of the Bethell structure are page 13, lines 16-18 teaching that the thickness of the ligand shell is determined by the size of the ligand molecule and page 24, lines 15-25 showing that ligand shell thicknesses on particles made with of alkanethiols having 6, 8, 12 or 16 carbons, C6, C8, C12 and C16, are 0.71 nm, 0.86 nm, 1.16 nm and 1.58 nm, respectively. This clearly shows that the thickness of the ligand shell of the particles is anticipated by the Bethell reference due to the use of similar compounds to synthesize the self assembled materials in a similar manner.)*

7. Claims 1- 5, 7 - 9, 21- 27 and 33 - 34 are rejected under 35 U.S.C. 102(b) as being anticipated by Anders (newly cited and applied). In the paper Andres teaches self-assembly of a two-dimensional superlattice of molecularly linked metal clusters. Close-packed planar arrays of nanometer-diameter gold clusters that are covalently linked to each other by rigid, double-ended organic molecules were self-assembled. Au nanocrystals, each encapsulated by a monolayer of alkyl thiol molecules were cast from a colloidal solution onto a flat substrate to form a close-packed cluster monolayer. Organic interconnects (aryl dithiols or aryl diisonitriles) displaced the alkyl thiol molecules and covalently linked adjacent clusters in the monolayer to form a two-dimensional superlattice of metal quantum dots coupled by uniform tunnel junctions. Electrical conductance through such a superlattice of 3.7-nm-diameter Au clusters, deposited on a SiO<sub>2</sub>

Art Unit: 1743

substrate in the gap between two Au contacts and linked by an aryl di-isonitrile [1,4-di(4-isocyanophenylethynyl)-2-ethylbenene], exhibited nonlinear Coulomb charging behavior. Page 1692 in the two paragraphs that bridge the three columns, ligand shell thicknesses of ~1.2 nanometers are taught for the dodecanethiol ligand shell along with the possibility that the molecules on adjacent clusters interpenetrate to give a cluster spacing that is less than the expected layer thickness. For the dithiol linked clusters the spacing is about 1.7 nm between clusters using a 2 nm length molecule as the linking molecule. In the last full paragraph of the center column of page 1691, Andres teaches that the synthetic method used produces particles that are equivalent to those produced by the Brust method (see reference 5). The advantage of using the Andres method is flexibility in the formation of the particles. Since Andres teaches particles of the correct diameter having a ligand shell of the proper thickness and means for measuring the electrical conductivity of the material, the claim elements are met and there is clear anticipation of the claimed structure.

8. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

9. Claims 2, 4, 6, and 22 are rejected under 35 U.S.C. 103(a) as being unpatentable over Bethell as applied to claims 1 and 22 above, and further in view of Natan (US 5,609,907). Bethell does not teach other types of metal colloids or the use of amine functions in the coating materials.

In the patent Natan teaches the formation of self-assembled metal colloid layers. Figure 1A with its associated discussion teach that the colloids can be gold, silver or other suitable metals. Column 3, lines 39 - 59 and the brief description of Figures 1A and 1B teach the additional use of amine and other functional groups in addition to thiols used to immobilize the colloids on a surface. The brief discussions of the figures also includes colloids having two layers of metal. Figure 1D shows the various levels of self-assembled colloids including multilayered (bulk).

It would have been obvious to one of ordinary skill in the art at the time the invention was made to incorporate the metal cores and metal interacting functional groups of Natan into the self-assembled colloid structures of Bethell because as shown by Natan the specifically claimed metals and functional groups would have been recognized as functional equivalents to those of Bethell relative to the formation of the self assembled colloid layers.

10. Claims 27 and 29-32 and are rejected under 35 U.S.C. 103(a) as being unpatentable over Bethell as applied to claims 1-5, 7-9, 21-26, and 33-34 above, and further in view of Terrill and Andres. Bethell does not teach the type of electrode used or the measurement equipment used.

In the paper Terrill discusses NMR, SAXS, Thermal, and Electron Hopping Studies of alkanethiol stabilized gold cluster monolayers in three dimensions. Au clusters stabilized by chemisorbed monolayers of octane-, dodecane-, or hexadecanethiolate were studied in solution and in the solid phase. These materials can be pumped free of solvent to form a dark brown solid that can be re-dissolved in nonpolar solvents. Their exceptional stability suggests that they may be viewed as cluster compounds. The self-assembled alkanethiolate monolayers stabilizing the metal clusters can be studied by using techniques that are insufficiently sensitive for study of a monolayer on a flat surface (e.g.,  $^1\text{H}$  and  $^{13}\text{C}$  NMR, elemental analysis, DSC, thermogravimetry (TGA), diffusion-ordered NMR spectroscopy (DOSY)). Results from such measurements (combined with SAXS data on solutions of the clusters and AFM and STM images) are consistent with a small, monodisperse (12 radius) Au core, which modeled as a sphere contains ~ 400 Au atoms and ~ 126 alkanethiolate chains, or if modeled as a cuboctahedral structure contains 309 Au atoms and ~ 95 alkanethiolate chains. High-resolution NMR spectra of cluster solutions display well-defined resonances except for methylenes nearest the Au interface; the absence of the latter resonance is attributed to a combination of broadening mechanisms based



Art Unit: 1743

on the discontinuous change in magnetic susceptibility at the metal-hydrocarbon interface and residual dipolar interactions. Films of the dry, solid cluster compound on interdigitated array electrodes (see pages 12538-12539, experimental section for preparation and measurements) exhibit current-potential responses characteristic of electron hopping conductivity in which electrons tunnel from Au core to Au core. The electron hopping rate decreases and the activation barrier increases systematically at longer alkane chain length. The results are consistent with electron transport rate control being a combination of thermally activated electron transfer to create oppositely charged Au cores (cermet theory) and distance-dependent tunneling ( $\beta = 1.2 \text{ \AA}^{-1}$ ) through the oriented alkanethiolate layers separating them. See pages 12545 - 12548 for a discussion of the electrical measurements.

In the paper Andres teaches self-assembly of a two-dimensional superlattice of molecularly linked metal clusters. Close-packed planar arrays of nanometer-diameter gold clusters that are covalently linked to each other by rigid, double-ended organic molecules were self-assembled. Au nanocrystals, each encapsulated by a monolayer of alkyl thiol molecules were cast from a colloidal solution onto a flat substrate to form a close-packed cluster monolayer. Organic interconnects (aryl dithiols or aryl diisonitriles) displaced the alkyl thiol molecules and covalently linked adjacent clusters in the monolayer to form a two-dimensional superlattice of metal quantum dots coupled by uniform tunnel junctions. Electrical conductance through such a superlattice of 3.7-nm-diameter Au clusters, deposited on a  $\text{SiO}_2$  substrate in the gap between two Au contacts and linked by an aryl di-isonitrile [1,4-di(4-isocyanophenylethynyl)-2-ethylbenzene], exhibited nonlinear Coulomb charging behavior. Page 1692 in the two paragraphs that bridge the three columns, ligand shell thicknesses of  $\sim 1.2$  nanometers are taught for the dodecanethiol ligand shell along with the possibility that the molecules on adjacent clusters interpenetrate to give a cluster spacing that is less than the expected layer thickness. For the dithiol linked clusters the spacing is about 1.7 nm between clusters using a 2 nm length molecule as the linking molecule. In the last full paragraph of the center column of page 1691, Andres teaches that the synthetic method used produces particles that are equivalent to those produced by the Brust method (see reference 5). The advantage of using the Andres method is flexibility in the formation of the particles.

Art Unit: 1743

It would have been obvious to one of ordinary skill in the art at the time the invention was made to incorporate the electrodes and measurement apparatus of Terrill into the formation method of Bethell because of their known use and sensitivity for measuring resistivity of self-assembled colloid clusters as shown by Terrill. It also would have been obvious to one of ordinary skill in the art at the time of the invention to use the ligand displacement method of forming the linked clusters in producing the layered structure of Bethell because of the greater flexibility in synthesis as taught by Andres

11. Applicant's arguments filed June 4, 2003 have been fully considered but they are not persuasive. Relative to the thickness of the coating in Bethell it should now be clear that the thickness is an inherent property since Bethell uses the same materials as in the instant specification to synthesize the particles by the same method as Bethell or a method which is equivalent to Bethell. The newly cited and applied Andres reference explicitly teaches that the coating of the particles form is within the claimed thickness. Therefore there is no question that this reference anticipates the claims. In response to applicant's arguments, the recitation of a chemical species has not been given patentable weight because the recitation occurs in the preamble. A preamble is generally not accorded any patentable weight where it merely recites the purpose of a process or the intended use of a structure, and where the body of the claim does not depend on the preamble for completeness but, instead, the process steps or structural limitations are able to stand alone. See *In re Hirao*, 535 F.2d 67, 190 USPQ 15 (CCPA 1976) and *Kropa v. Robie*, 187 F.2d 150, 152, 88 USPQ 478, 481 (CCPA 1951). Relative to this examiner points out that the interaction with a chemical species is an inherent property of the material being used to interact with the species. Thus since the same materials are taught by Bethell and Andres as found in the instant specification, the body of the claim does not depend on the recitation of a chemical species for completeness and the limitations are able to stand alone. Applicant is directed to claim 1 relative to arguments about the sensor being an electrical chemical sensing device. Claim 1 is of a scope that encompasses much more than electrically sensing a chemical of interest. Relative to the recitation of a sensor, page 1, line 19 to page 17, line 7 teach both optical and electrical properties being measured. Furthermore page 19 lines 3-12 teach a battery and a current meter as the equipment necessary to measure (sense) the

electrical conductivity to determine if change has occurred. Therefore this is the scope examined in the interpretation of the general recitation of a sensor as found in most of the claims.

The combination including the Natan reference is used to show that other types of ligands and metals can be used to form the particles. The obviousness is based on the similarity of the methods and the final products. The claims being rejected by the combination are generally the article of manufacture rather than the sensor claims. Thus the claims are directed toward an article that could have several uses such as an optical sensor. This is consistent with the instant specification as noted above. Thus the combination of Bethell with Natan is a proper combination showing the obviousness of substituting the materials of Bethell with other materials.

Relative to the combination including the Terrill reference applicant is first directed to claim 24 which sets a film thickness of 5 to 10,000 nm (0.005 – 10  $\mu$ m). Thus about half of the range of the film thickness taught by Terrill is within the specifically claimed thickness. However claim 24 is a dependent claim which does not limit the thickness of any claim that does not depend therefrom. With respect to all claims not having the thickness limitation, the argument is not commensurate in scope with the claims. Even with claim 24 it is not persuasive since there is a clear overlap in the ranges taught and claimed. Again relative to the method of forming the material through spraying, the claims are not limited to a material produced by that method and the argument is not commensurate in scope with the claims. Relative to the layering method, the Andres reference clearly shows the difference between the method of Bethell and the claimed method to be obvious because of the flexibility offered using a ligand displacement method.

12. Claims 35-36 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims. The art of record fails to teach or suggest the claims combination or elements. It is noted that although the respective elements are conventional as evidenced by the page 21, line 17 to page 22, line 8, examiner was not able to determine if they are part of the measurement apparatus taught by Terrill.

Art Unit: 1743

13. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. The additionally cited art relates to sensor structures and nanoparticles having an organic ligand coating.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arlen Soderquist whose telephone number is (703) 308-3989. The examiner's schedule is variable between the hours of about 5:30 AM to about 5:00 PM on Monday through Thursday and alternate Fridays.

For communication by fax to the organization where this application or proceeding is assigned, (703) 305-7719 may be used for official, unofficial or draft papers. When using this number a call to alert the examiner would be appreciated. Numbers for faxing official papers are 703-872-9310 (before finals), 703-872-9311 (after-final), 703-305-7718, 703-305-5408 and 703-305-5433. The above fax numbers will generally allow the papers to be forwarded to the examiner in a timely manner.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is (703) 308-0661.



August 22, 2003

ARLEN SODERQUIST  
PRIMARY EXAMINER